Synthetic Organic Chemicals

PUBLISHED BY THE

EASTMAN KODAK COMPANY, ROCHESTER, NEW YORK

VOL. VI DECEMBER, 1932 No. 2

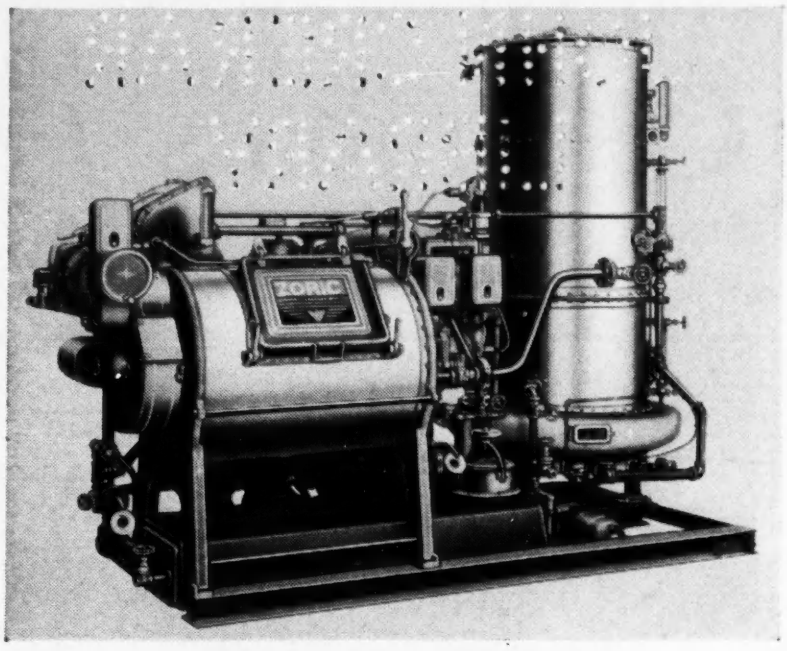
Organic Chemicals in the Dry-Cleaning Industry

THE dry-cleaning industry is one which has made rapid strides in the last few years as a result of applying scientific methods of research to its problems. It was only a short time ago that most of the dry cleaning was done in small shops with practically all of the operations carried out by hand. There are now over 5,000 power dry cleaning plants in the United States and Canada doing a total yearly business of approximately $500,000,000.

The process of dry cleaning is so well known that no general description is necessary. It consists simply in extracting soluble dirt, as well as suspended particles, with a non-aqueous solvent. This avoids possible shrinkage, warping, or injury to the finish of goods that might take place when laundered in the usual manner with soap and water. Ordinary clothing, draperies, and so forth, are placed in a rotating washer and treated either continuously or intermittently with the dry-cleaning fluid. Less rugged materials are cleaned by hand. The excess solvent in some cases is removed centrifugally before drying in a chamber ventilated by a current of warm, dry air.

The early dry-cleaning fluids consisted almost entirely of gasoline which had many disadvantages, not the least being the fire hazard. Through the cooperation of the National Association of Dyers and Cleaners with the Mellon Institute, a petroleum distillate was found which quickly superseded gasoline. The specifications of this new product, named "Stoddard Solvent," after one of the originators, were chosen to fit ideal requirements as closely as possible. The high flash point, over 100° F., removes danger from static ignition. Freedom from corrosive substances protects metal equipment, while absence of unsaturated hydrocarbons lowers the tendency to add impurities. The distillation range selected prevents waste from evaporation of low-boiling fractions yet it is not high enough to interfere with complete removal of solvent on drying. A clean, sweet odor results from the thorough exclusion of sulfur-containing compounds.

The use of chlorinated hydrocarbons has also progressed quite rapidly in this country in the last few years. Although they are practically non-flammable, their cost is higher than that of the standard petroleum solvent, and there has been some question as to their physiological action. With the use of completely enclosed systems, however, the toxic or anesthetic effect of the chlorine hydrocarbons is not important nor is the volatility a disadvantage.



A modern dry-cleaning unit

Sulfur-free carbon tetrachloride and stabilized trichloroethylene are both claimed to be particularly good solvents. They completely remove many of the oily stains encountered that are not readily extracted with petroleum solvents, and which ordinarily must be "spotted out" later.

Recovery of the cleaning fluid is essential as its cost is a large factor. This is especially true since it is necessary to make frequent changes of the solvent in order to keep the finished goods bright in appearance. The need for this is readily apparent from the estimate that the average soiled suit of men's clothing contains about 2½ ounces of extractible material.

In the case of the naphtha solvent, several methods of repurification or recovery are in use. Centrifuging will separate the solid particles, but after two or three treatments enough oily material remains in solution to darken the solvent and prevent its continued use. Treatment of the used liquid with diatomite and running it through a filter press is a little more efficient and allows a longer use of the solvent. In both methods, however, soluble materials accumulate and must be removed by vacuum distillation, often preceded by treatment with alkali. Agitating the solvent with caustic soda saponifies the fatty oils and greases and precipitates the sodium soaps which can then be filtered off. Final washing with water removes any residual alkali, soap, or other water-soluble impurities.

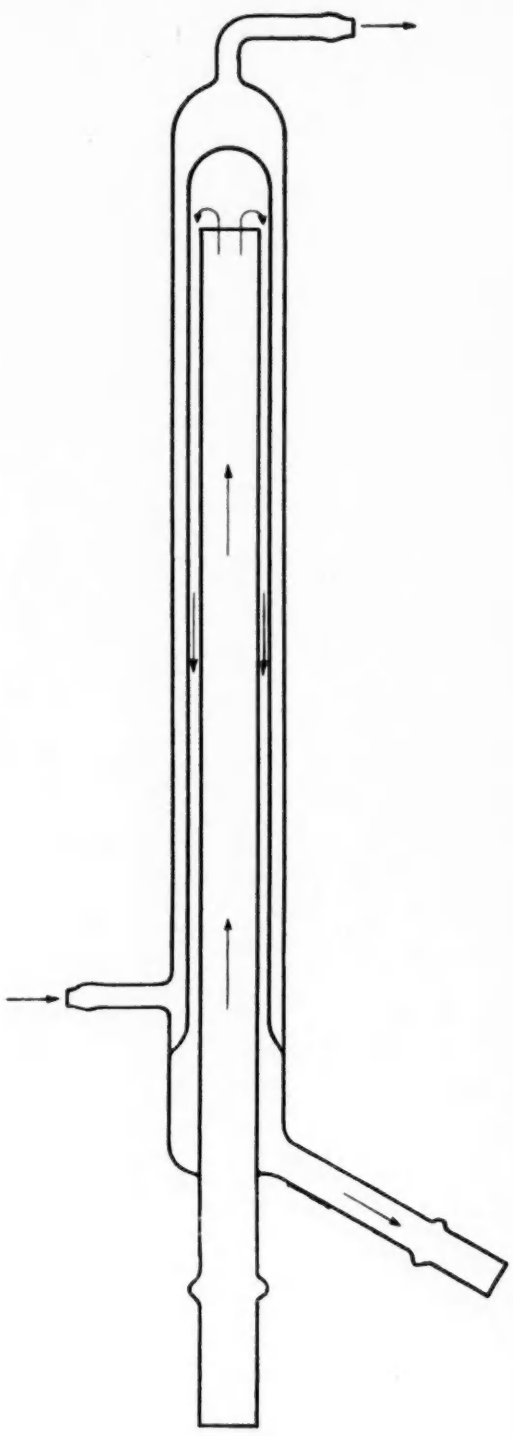
In most cases the chlorinated solvents are filtered and distilled without any chemical treatment, and in some types of equipment the entire operations of cleaning and recovery of solvent are carried out in the one enclosed unit. The accompanying illustration, obtained through the courtesy of the American Laundry Machinery Company, shows one of their self-contained dry-cleaning outfits, designed primarily for clothing.

It is interesting that the cleaning with both types of solvents is improved by the addition of small quantities of soluble soaps. Ordinary laundry soaps are insoluble in cleaning fluids but there are a number of "benzine soaps" available for dry cleaners. One of the most efficient is potassium oleate containing a slight excess of oleic acid. This is prepared by dissolving caustic potash in methyl alcohol and stirring it into the liquid fatty acid. A soap concentration as low as 1 part in 400 will make a distinct improvement in the cleaning liquid, both as to time required and results obtained. On the contrary, too much soap lessens the cleaning effect. Soaps made from triethanolamine instead of caustic potash are used also. Other chemical products that are added to improve the detergent properties are ammonia, cyclohexanol, and pine oils.

Since 1927, the National Association of Dyers and Cleaners has maintained a laboratory at Silver Springs, Maryland, for research on dry-cleaning problems. A vocational school where members can obtain training is also located there.

A Water-Cooled Still Head

THE type of condenser shown in the photograph and drawing reproduced on this page offers many advantages in general laboratory use. The rate of distillation is greatly increased as compared with that of the ordinary goose-neck head connected to a Liebig condenser. Since the cooling takes place on the side-wall only, the inner tube stays at the temperature of the vapor. As a result condensation does not take place within the center tube to slow up the distillation by the unwanted refluxing action.

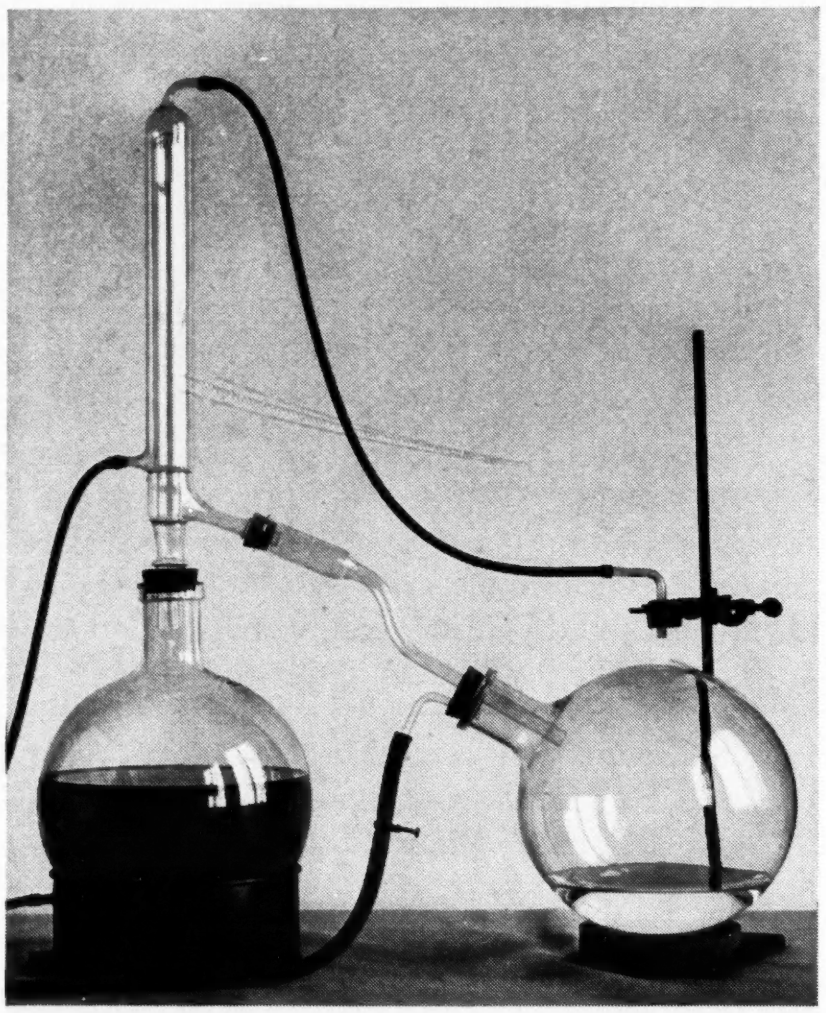


The value of this still head is readily appreciated when it is necessary to concentrate large volumes of liquids by removing the solvent. Distillations under vacuum can be carried out very easily, an example being the removal of water from benzene or toluene by distilling off the azeotropic mixture. The illustration below shows the purification of thionyl chloride by distillation, after treatment with linseed oil to remove the color.

The head itself is really quite rugged as it does not need to be more than fifteen to eighteen inches in height. It can be quickly set up or dismantled since no clamps or ringstand are required for support. Any one accustomed to making inner seals will have little trouble with the glass blowing.

LEFT—*Diagram showing construction of still head and path of vapor.*

BELOW—*Distillation set-up for removing colored impurities in thionyl chloride.*



Eastman Organic Chemicals as Analytical Reagents

XXVII Reagents for Titanium.

CUPFERRON

Von Bergkampf, Z. ANAL. CHEM. *83*, 345 (1931)

Titanium, iron, and aluminum can be separated when in an acid tartrate solution. While still acid, the solution is treated with hydrogen sulfide to reduce the iron to ferrous condition. Further treatment with hydrogen sulfide, after adding an excess of ammonia, precipitates the iron as ferrous sulfide. The filtrate is made acid with sulfuric acid and the titanium precipitated in the cold with 6% cupferron solution. Aluminum remains in solution. Vanadium and zirconium interfere with the separation.

8-HYDROXYQUINOLINE

Berg and Teitelbaum, Z. ANAL. CHEM. *81*, 1 (1930)

Solutions of titanium in the form of its chloride are treated with small amounts of tartaric acid and sodium acetate, then neutralized with ammonia. After adding a few cubic centimeters of glacial acetic acid, the solution is heated to 60° C. and the titanium precipitated with 8-hydroxyquinoline dissolved in alcohol. The orange-yellow crystalline compound is filtered, washed with hot water, and dried at 110° C. It contains 13.61% titanium.

5.7-DIBROMO-8-HYDROXYQUINOLINE

Berg and Küstenmacher, Z. ANORG. CHEM. *204*, 215 (1932)

This compound can be used as a specific reagent for titanium, copper, or iron as it produces a colored precipitate with all three in strongly acid solutions. The reagent in water-acetone-acid is added to a hot solution of the metal. The titanium precipitate is formed when present in amounts as small as 0.1 mg. in 100 cc. Under the proper conditions, titanium can be separated in the presence of copper, iron, mercury, chromium, and aluminum.

TANNIC ACID

Powell and Schoeller, ANALYST, *55*, 605 (1930)

Titanium forms a vivid red precipitate with tannic acid which can be used to separate it from aluminum, zirconium, and thorium. A double precipitation of this complex from a neutralized oxalate solution, half saturated with ammonium chloride, separates titanium from zirconium completely and quickly. The reagent is a freshly made and filtered 4% solution of tannic acid in water.

Twenty-five New Eastman Organic Chemicals

The following chemicals have been added to our list since the last issue of this bulletin. Purity specifications and prices of these compounds will be supplied promptly on request.

Acetone Phenylhydrazone
Acetyl-m-Anisidine
Acetyl-o-Anisidine
N-Benzylpyrrole
Bromomesitylene
iso-Butyryl Chloride
p-Chlorobenzoyl Chloride
Crotonyl Chloride
2.4-Dibromo-α-naphthol
s-Di-p-nitrophenylcarbazide
β-Dithiodiglycol
o-Ethoxybenzaldehyde
p-Ethoxydiphenyl
Ethyl n-Butyl Sulfide
Ethyl Crotonate
m-Fluorochlorobenzene
Guaiacol Benzoate
n-Heptyl Iodide
Methyl n-Butyl Sulfide
Naphthoresorcinol
3-Nitroacenaphthene
o-Nitrobenzhydrazide
p-Nitrobenzhydrazide
β-Thiodiglycol
Trithiomethylene